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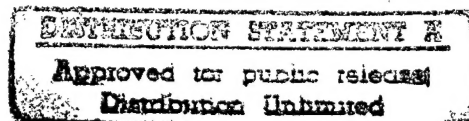
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THE BENEFICIATION OF NICKEL OXIDE ORES
(USSR)

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FOREWORD

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THE BENEFICIATION OF NICKEL OXIDE ORES
(USSR)

(Ye. K. Zagirova and Tsi Chzhi-Chzhon took part in this work)

Tsvetnyye Metally
/Ferric Metals/,
No 11, November 1959, Moscow,
Pages 18-25,
Russian, per

G. A. Khan, A. I. Zarakhani

At the present time nickel oxide ores are refined by reduction-sulfidization smelting in shaft-type furnaces. The immediate melting of the entire mass of ore requires a high quantity of coke (up to 40 tons for one ton of nickel) and gives a low yield of metal (on the order of 65%). Therefore, for the sake of economy it is expedient to utilize for this purpose only comparatively rich ores, containing over 0.8% Ni. For the economical refining of poorer grade ores it is necessary to beneficiate them beforehand.

However, basic investigations on the beneficiation of nickel ores prove that these ores do not yield to the usual methods of concentration.

Investigations [1, 5, 6] show that in such ores nickel only occurs in the form of isolated minerals (garnierite, nickel-bearing minerals, etc.). A considerable part of the nickel is replaced isomorphically by other cations in various silicates or is absorbed by them. On the basis of a study on the solubility of ores and their ingredients in various solvents it was determined that nickel is chiefly connected with iron- and aluminum-magnesium compounds and only part of it is found in the form of a free nickel silicate.

During experimentation with the beneficiation of ores [1] the most diverse methods of flotation were tried, and many flotation reagents tested. However, no positive results were obtained. Hydro-metallurgical methods (with the exception of ammonium leaching of the concentrated ore) were also not applicable, chiefly because of the high consumption of the solvent.

Because of this it was necessary to find other means of beneficiation of ores. One of the methods was beneficiation with a preliminary pyrometallurgic preparation of ore.

As a preliminary operation in the beneficiation, reduction of the ores was undertaken [2, 3]. The reduced material was then subjected to magnetic separation flotation, and leaching. Not one of these methods, however, yielded a sufficiently high extraction of recovered nickel.

In his time, G. A. Shakhov [4] proposed a preliminary sulfidization of nickel oxide ore with subsequent flotation of the sulfidized material. However, with the utilization of ferrous sulfide for this purpose in the absence of a reducing agent the degree of sulfidization amounted to 50%, whereas with the utilization of the vapor of elemental sulphur all of the iron was sulfidized completely along with the nickel. Therefore, flotation indices were very low.

In 1952, in Mintsvetmetzoloto [Moskovskiy Institut Tsvetnykh Metallov i Zolota--Moscow Institute of Nonferrous Metals and Gold] along with V. A. and A. V. Vanyukov, under the leadership of A. V. Vanyukova [7] we carried out a more detailed investigation on the sulfidization of nickel oxide ore and its subsequent flotation. The results revealed that the preparation of the ore must not only include the transformation of the oxide compounds of nickel into sulfides, but the crystallization of the obtained sulfides into sizes which insure their extraction during flotation.

From a number of methods tried for the preparation of nickel oxide ore for flotation, the most successful was the method of preliminary reduction of the ore, with subsequent sulfidization with pyrite in a cylindrical rotating kiln and coarsening [in size] of the obtained sulfides.

The tests were performed with low-grade nickel oxides from the Aydarbaskian deposit, containing 0.74% Ni, in the laboratory with a cylindrical rotating kiln.

The higher ferric oxide was reduced to ferrous oxide with gaseous ($\text{CO}_2 + \text{CO}$) and solid (coke dust) reducing agents.

The reduced material was subjected to sulfidization in a nitrogen atmosphere at 1000-1200°.

From microscopic investigations it was determined that the nickel in ore sulfidized in this manner rarely occurs in form of pentlandite but is most frequently associated with pyrrhotite and metallic ore forming solid solutions. Complex sulfide mixtures are also encountered, composed of pyrrhotite, metallic iron, and magnetite.

The composition and structure of the treated material change considerably, depending on the conditions of the sulfidization. The ratio of pentlandite to pyrrhotite changes with the size of the inclusions.

In the coarse particles pentlandite predominates; in the finer ones, pyrrhotite predominates. The degree of coarseness of the sulfides depends on the temperature and cooling time. With the lowering of the temperature from 1200 to 1000° the extraction of nickel into the concentrate is decreased; this is explained by the fact that sufficient mobility in the softened sulfide mass takes place only at 1200°.

The technological indices of flotation are influenced by the consumption of pyrite during sulfidization. With an increased consumption of pyrite (from 10 to 15% of the weight of the initial ore) the extraction of nickel into the concentrate is increased, but the quality of the concentrate drops.

On the basis of these laboratory investigations this Institute, along with the workers of the Yuzhuralnikel Combine Yuzhno-Ural'skiy nikel'nyi zavod., -- South Ural Nickel Plant in Orsk/, carried out semi-commercial investigations to sulfidize ore with pyrite and reduce it directly with coke dust and Kuznetsk coals in the presence of phosphate rocks and NaF, and also without them, and the subsequent refining of the prepared product.

In order to carry out these tests, the experimental refining furnace of the Orsk-Khalilovskiy Metallurgic Combine was used composed of two separate parts: the reduction part (7.85 x 1.04 m) and the refining part (2.5 x 0.96 m), linked by a reloading attachment. The kiln was heated with coking gas. The temperature in the reduction zone was 900-950°, in the sulfidizing zone, from 1160-1180°.

Because of the absence of a constantly working coarsening concentration apparatus the medium sized samples of the preliminary material were subjected to testing for concentration under only laboratory conditions.

A comparison of the test methods of pyrometallurgical preparation is shown in Table 1. First of all, the reducing sulfidizing roast was tested, which in principle can be done during the extraction of the highly metalized phase with minimum sulphur content, as well as during the extraction of the sulfide phase, which scarcely contains any metal at all. This is achieved by the change of the ratio of the reducing agent and the sulfidization agent in the feed. Basically, these two methods were tested.

The tests were carried out with submarginal ores of the Aydarbaskiy deposit, as well as with ores taken from Kimpersaysk, Akermanovsk, and the Buruktal'sk deposits with a commercial nickel content.

Table 1.

A Comparison of the Method of Pyrometallurgic Preparation of Ores

Type of ore	Working Method	Composition of matte Inclusions	Method of Oxidation	Yield in %	
				Concentrate*	Residue
Kimpersaysk Deposit	Coke dust	Pentlandite	magnetic separation and flotation	7.98	15.43 84.57
	10-15% pyrite 10-15%	70-85% Metallic Phase 5-15%		7.45	
Kimpersaysk Deposit	Coke dust 3%	Pentlandite 20-40%	Flotation and	11.31	14.74 85.26
	pyrite 15%	pyrrhotite 50-70%. Metallic phase 5-15%	magnetic separation	3.43	
Akermansk Deposit	Coke dust	Pentlandite 30-50%	magnetic separation and flotation	14.82	26.38 73.62
	30-40% Pyrite 5% Limestone 5%	pyrrhotite 10-70% metallic phase 30-50%		11.56	
Buruktal'sk Deposit (Magnesia)	Coke dust 20%	Metallic phase 6.74-7.1% Ni	Wet magnetic separation on a magnetic separator	11.91	88.09
Buruktal'sk Deposit (Magnesia)	Coke dust 20%	Metallic phase 10.95-16.37% Ni	Dry magnetic separation on a disc separator	9.73	90.27
	phosphor 5%				

* Upper figure is the sum of concentrates and flotation -- lower the intermediate product.

Table 1 (Continued)

Type of ore	Working Method	Composition of matte Inclusions	Method of Oxidation	Yield in %	
				Concentrate*	Residue
Buruktal'sk Deposit (Magnesia)	Coke dust 20% NaF 4%	Metallic phase 8.83-2% Ni	Wet magnetic separation on drum type separator	15.7	84.30

Buruktal'sk Deposit (Magnesia)	Coal Kuznetsk 25% Phosphate rock 5%	Metallic phase 11.25 - 14.65% Ni	Wet magnetic separation on drum type separator	18.77	91.10

Table 1. (Continued)

Type of ore	Content in %				Extraction in %			Remarks
	Sinter		Concentrate*	Residue	Concentrate*		Residue	
Kimpersaysk Deposit	1.51	14.40 2.36	8.4	0.25	74.32 11.66	85.98	14.02	
Kimpersaysk Deposit	1.37	9.5 1.61	7.57	0.30	77.32 4.03	81.35	18.65	
Akermansk Deposit	1.96	10.90 2.24	7.1	0.12	82.38 13.13	95.51	4.49	
Buruktal'sk Deposit (Magnesia)	1.05	8.21		0.088	92.85	7.15		Thickness of initial material - 0.58 mm strength of magnetic field 765 oersteds
Buruktal'sk Deposit (Magnesia)	0.92	8.36		0.122	88.1	11.90		Size of initial material 1 mm; H of basic separation 2038 oersted; H processing 640 oersted. Wet separation on drum separator of the given example was not carried out.

Table 1. (Continued)

Type of ore	Content in %		Extraction in %		Remarks
	Sinter	Concentrate* Residue	Concentrate*	Residue	
Buruktal'sk Deposit (Magnesia)	0.83	4.87	0.08	91.91	8.09 Size of initial material 1 mm; H = 765 oersted

Buruktal'sk Deposit (Magnesia)	0.87	4.24	0.096	91.10	8.9 H = 765 oerst.

The consumption of coke dust changed from 3 to 40%, and pyrite from 3 to 15%, of the weight of the original ore. In individual batches 5% limestone was added in order to decrease the temperature of the smelted ore in the feed.

Microscopic investigation [See Note] revealed that in all ore samples, including those fired with various admixtures as well as those not containing admixtures, and in the beneficiation products, two sharply different components are found: the slag and the matte. The first one has the principal amount of clinkering capacity and has a relatively constant composition, depending little on the makeup of the kiln. In it glass and pyroxenes, $(\text{Fe}, \text{Mn})\text{SiO}_3$, predominate; also, white quartz, chrome spinellides, and coke are found. In the second component all elements are found which comprise the metallic phase and the sulfides. In a number of cases the sulfides are generally absent in the sinter. The matte part consists of ferronickel (Fe, Ni) with a variable content of iron and nickel, pyrrhotite with an admixture of nickel, and pentlandite in the form of separate deposits or thin blades which are formed during the decomposition of pyrrhotite. ([Note] Microscopic investigations were carried out by A. Yu. Malevskiy.)

In carrying out the process for the obtaining of the sulfide phase, even with a minimum consumption of the reducing agent (coke 3%), in the matte comprising the sinter, from 5 to 15% of the metallic phase is combined, therefore a combined method of beneficiation was adopted; flotation and magnetic separation. Depending on the ratio of pyrite and coke in the feed, the magnetic separation is utilized either in the beginning of the test or at the end.

With the consumption of 10-15% coke and the same amount of pyrite in the matte, the metallic phase occurs in the form of large buttons which are difficult to extract by flotation; therefore, in this particular method magnetic separation preceded flotation. In such sequence of the operations approximately 43% nickel is extracted into a magnetic concentrate containing 14.3% Ni. About 31% nickel is extracted into 13% nickel flotation concentrate. The total extraction of nickel in the concentrate and intermediate product with a mean weight content of 8.4% Ni amounts to approximately 86%.

Magnetic separation was carried out with the aid of a hand magnet,³ whereas the flotation was done in a laboratory flotation machine 750 cm³ in volume, solid : liquid ratio = 1:3, in two stages. The fineness of the millings before the first stage is 50-60% less than 0.074 mm; before the second one it is 90-95% less than 0.074 mm. The consumption of butyl xanthate amounts to 200-300 and pine oil 120-130 g/t. Such an increased consumption of reagents is conditioned by the presence in the sinter of coke dust.

In lowering the coke dust content of the feed to 3%, a finer impregnation of matte particles is obtained, and therefore flotation has to be utilized as the principal method of beneficiation. The quality of the flotation concentrate with such a consumption of coke dust is lowered somewhat (9.5-10%) apparently as a result of the lowered pentlandite content in the matte comprising the sinter, 70-85 to 20-40%. With such a method of preparation almost the entire metallic phase changes into a flotation concentrate, and the extraction of nickel increases to 77.3% in this. However, the magnetic separation remains as a controlling operation and is used for catching coarse pieces. In most experiments the magnetic concentrate comprises an intermediate middling product containing approximately 2% Ni. The total extraction of the concentrate with an average weight content of 7.57% Ni amounts to 81.3%.

In carrying on the process for obtaining the metallic phase, the coke-dust amounted to 20-45% but pyrite was 5%. In order to lower the smelting temperature of ore in the feed, 5% of limestone was added. The sinter thus obtained differed in the variability of composition of the matte inclusions, the metallic phase amounting to 10-16%; pyrrhotite 10-70%, and pentlandite 20-50%.

With such a regimen of ore preparation, the combined method (magnetic separation and flotation) yields good technological results. Sixty-five to 82% of the nickel is extracted into the magnetic concentrate, containing 8-11% Ni. Additionally, 13-25% nickel is extracted, yielding a concentrate containing 3-4% Ni. The tailings contain 0.10-0.15% Ni, while the sinter contains 1.3-1.5% Ni. The total extraction of nickel into the concentrate and middling which has to be returned to the furnace, amounts to 90-95%.

In addition a dried reduction of the ore without the addition of pyrite with a 20% of consumption of coke dust and 5% of limestone was tried. The concentration of the sinter was carried out by magnetic separation. The controlling flotation of the residue by magnetic separation did not give positive results. The extraction of nickel in the magnetic concentrate amounted to 80-87% at a 9-12% Ni content. The nickel content in the sinter was 0.8-1.5%.

Tests have shown that in changing the consumption of the reduction agent a considerable yield of the metallic phase with a high cobalt content may be obtained.

Taking into account completely satisfactory indices of magnetic separation of the sinter, obtained without the addition with sulfides, further investigations were carried out with the object of selecting the optimum means of reduction, method of beneficiation and type of magnetic separation. The contents of the tested feeds are shown in Table 2.

Table 2.

The Contents of the Feed, % of Ores

<u>Coke Dust</u>	<u>Kuznetsk Coal</u>	<u>Phosphate Rock*</u>	<u>Sodium fluoride*</u>
20	--	--	--
20	--	5	--
20	--	--	5
--	25	5	--

* Phosphate rock and sodium fluoride were used in order to improve conditions for enlarging the metallic particles.

During the pyrometallurgical preparation of ores without a sulfidizer, the sinter particles are composed only of a ferronickel solution with variable contents of iron and nickel. The inclusions in the slag phase are differentiated in size as follows: large residues 0.35 - 5 mm in size, and small inclusions from 0.02 mm and smaller (the average size of inclusions was 0.003 - 0.010 mm). The fine inclusions are concentrated in small areas 0.15-1.5 mm in size, where its density reaches 5-10%. In the rest of the slag single inclusions occur, the quantity of which on the whole does not exceed 1-2%. The large grains from 0.1-5 mm. i.e., the basic mass of metal (90-95%) are rounded (Figure 1). Such inclusions are localized chiefly near the large pores and closer to the surface. In samples which were fired without additives, the metallic inclusions have a dendritic, worm-like form (Figure 2). Similar separations are closely connected with slag and are frequently reminiscent of graphic intergrowth.

During the thermal preparation of ores without pyrite additives, a great quantity of iron is reduced, in connection with which the nickel content in pure metallic residues fluctuates within the wide limits of 2-16.3%. Therefore, with the high content of iron in the ore and the application of this method for thermal preparation it is not possible to obtain a high nickel concentrate.

In examining the technological results of the concentration of the indicated samples on a wet magnetic drum-type separator (see Table 1) it follows that the reducing roast with the application of coke dust only of 20% quantity, gives approximately the same technological results as a reducing-sulfidizing roast, with a 5% addition of pyrite. The addition of phosphate rock into the feed almost doubles the nickel content in its metallic phase, however, it does not better the technological results. In exchanging phosphate rock with sodium fluoride (5% consumption) the extraction of nickel increases with a simultaneous lowering of the quality of the concentrate by about half. The replacement of dust (coke) with Kuznetsk coal increases the extraction of nickel by 2%, but its contents drops to 4%.

Table 3.

A Comparison of the Concentration Methods of a Sample of the Buruktal'skiy Ore

Method of Enrichment	Yield, %		Content rate, %			Extraction, %		Strength of magnetic field oresteds.
	Concen- trate	Resi- dues	Sin- ter	Concen- trate	Resi- dues	Concen- trate	Resi- dues	
Jigging*	3.04	72.45	0.58	13.3	0.17	69.9	21.3	--
Concentration on the table	13.96	86.04	0.85	5.29	0.126	87.2	12.80	--
Wet magnetic separation on a roller separator AX-1	19.44	80.56	0.84	3.95	0.096	90.86	9.14	700
Wet magnetic separation on a drum-type separator	15.70	84.35	0.83	4.87	0.03	91.91	8.09	765
Dry magnetic separation on a disc separator	16.45	83.55	0.84	4.60	0.101	89.97	10.03	1590 640

* The middlings contain 0.21% Ni. The nickel extraction in it amounts to 0.83% (with the middlings comprising 24.5%)

Finally, it has to be pointed out that from the investigated methods of pyrometallurgic preparation the highest technological indices were obtained with a reducing sulfidizing roast (the consumption of coke dust is 30%, pyrite 5%, and limestone 5%). It should be pointed out that during such experiments a slightly higher nickel bearing sinter was obtained, containing 1.96% Ni. Along with this method it is possible to employ ore reduction processes which involve the use of coke-dust only-consumption up to 20% -- or a mixture of coke dust with phosphate rock of sodium fluoride.

During investigation of beneficiation of the samples, fired with or without additions of phosphate rock and NaF, the possible application of various methods of concentration were studied: jigging, concentration on a table; magnetic separation and a combination of these methods.

Comparison of the beneficiation methods for samples of magnesium ores of the Buruktal'sk deposit, roasted with 20% coke dust and 4% NaF, and samples of ores from the Kimpersaysk deposit, roasted with 5% phosphate rock, is illustrated on Table 3.

Data in Table 3, as well as tests carried out with other samples, show that the most effective way (method) of concentration of the reduced ore is wet magnetic separation on a separator with a weak magnetic field ($H = 765$ oersteds).

In coarse material -- 1 mm -- a high extraction of nickel, about 91% with a 4.87% concentration in its contents, was obtained. In the direct magnetic separation without supplementary operations more than 84% of waste residues were obtained with a 0.08% Ni content. The extraction of cobalt by such a process amounts to 82% with a 9.3% concentration in its content.

Concentration on a concentration table and jigging on a reciprocating jig yielded a lower extraction, since with the gravitational method of concentration a richer residue is obtained due to the free metallic particles 0.02-0.03 mm in diameter and 0.01 mm thickness which are extracted through magnetic separation. Besides, during magnetic separation slag particles with finely dispersed inclusions of the metal are extracted, due to the increased magnetic attraction. In the gravitational method such particles are not extracted, since the finely dispersed inclusions do not influence the specific weight of the slag.

Conclusions

1. The possibility of beneficiating nickel oxide ores has been determined by the flotation method and magnetic separation, with preliminary pyrometallurgic preparation either by a reducing-sulfidizing roast with the addition of coke dust and pyrite, or by a direct reduction with coke dust in the presence of phosphate rock or sodium fluoride.

In utilizing the reduction sulfidizing roast it is necessary to employ a combined method of concentration -- flotation and magnetic separation. In carrying out the reducing roast without the addition of pyrite, the basic process of concentration is wet magnetic separation. Utilizing this scheme in order to separate coarse residues up to 1-2.5 mm liberated during the refining, it is expedient to include jigging or hydraulic classification.

2. Enriching the nickel oxide ore with a content of 0.8 - 1.5% Ni, prepared according to the pyrometallurgic method, a concentrate with a 7-8% Ni content may be obtained with an extraction of 88-95%. With lower extractions on the order of 75-82%, a concentrate of 11-14% Ni may be obtained.

3. The nickel content in the concentrate basically depends on the quantity of the reduced iron in the sinter. The more iron found in the sinter, the lower is the nickel content in the concentrate; the higher, however, is its extraction.

The investigations carried out permit us to affirm that the problem of the refining of lean nickel oxide ores may be solved in a positive way.

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FIGURE APPENDIX

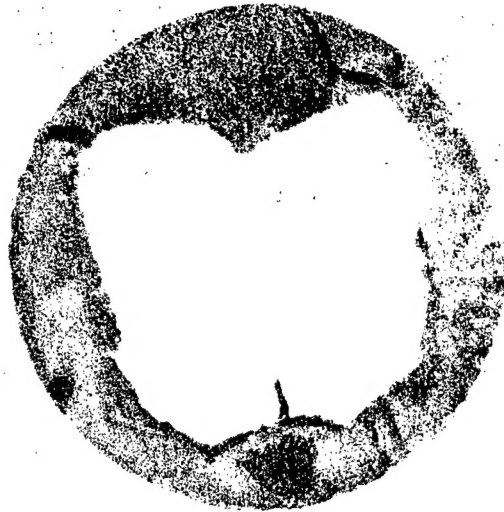


Figure 1. Inclusions of large grains of ferro-nickel (white) in silicate (grey)

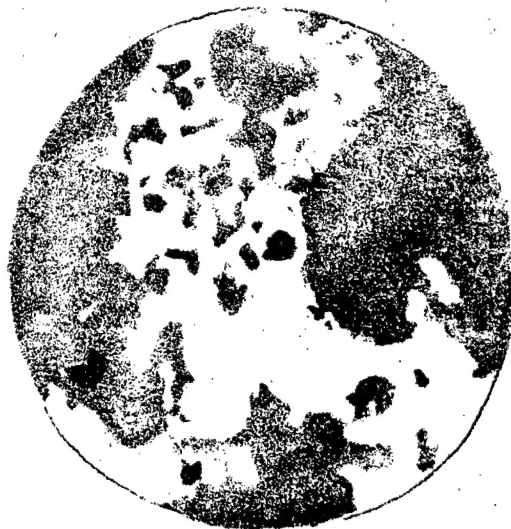


Figure 2. Penetration of ferro-nickel (white) in silicates (grey).